



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the application of

Deborah Tung, Edwin A. Sisson,  
and Roy A. Leckonby

Serial No. 09/916,671

Filed July 26, 2001

For OXYGEN-SCAVENGING RESIN  
COMPOSITIONS HAVING LOW  
HAZE

Examiner Tae H. Yoon  
Group Art Unit: 1714

**Certificate of Mailing**

I hereby certify that this correspondence was deposited with the United States Postal Service as first class mail in an envelope addressed to: Mail Stop Non-Fee Amendment, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on this 20<sup>th</sup> day of August, 2003.

*Cynthia M. Wilson*  
Cynthia M. Wilson, Sec'y to Rodney L. Skoglund

**DECLARATION OF EDWIN A. SISSON**

**37 C.F.R. § 1.132**

Sir,

As the inventor of the subject application, I hereby declare that:

1. I am a resident of Medina, Ohio and am an inventor of the subject application.
2. My educational background includes a Bachelor of Science degree in Chemical Engineering from the University of Nebraska (1985), a Bachelor of Arts degree in Communications from the University of Nebraska (1985), and a Masters of Business Administration from Case Western Reserve University (1991). I am a named inventor in eight United States letters patents in the field of polyester polymers.
3. I have been employed by M & G USA Corporation and its predecessors for approximately twenty years and have worked in the field of polymer resins for seventeen years. I am presently Manager, Resin Development at M & G USA Corporation's Sharon Center, Ohio research facility.
4. I reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6, 10, 11, 34, 36, 37, 39,

and 42-44 under 35 U.S.C. § 102(b) as anticipated by, or in the alternative, under 35 U.S.C. § 103(a) as obvious over Nichols, U.S. Pat. No. 5,008,230 (the '230 patent). I respectfully disagree.

5. In my experience, I have overseen many experiments where catalysts of metal salts, such as those taught by the '230 patent, are not oxygen-scavenging particles. The '230 patent teaches a catalyst system for producing polyethylene terephthalate (PET). (Col. 2, lines 36-38). The catalyst system includes (a) antimony; (b) cobalt or zinc; and (c) zinc, magnesium, manganese, or calcium. (Col. 2, lines 44-49). More particularly, the '230 patent teaches that suitable catalyst compounds are "in the form of inorganic compounds such as carboxylates (such as acetates), compounds of Group VI (such as oxides or sulfides), halides such as chlorides, amines, ... ." (Col. 4, lines 37-41). Table 1 below illustrates the lack of oxygen scavenging of resins comprising the catalyst composition taught by the '230 patent.

TABLE 1

	SAMPLE 1	SAMPLE 2	SAMPLE 3
ANTIMONY GLYCOLATE (ppm Sb)	0	600	600
COBALT ACETATE (ppm Co)	0	60	60
ZINC ACETATE (ppm Zn)	0	150	0
MAGNESIUM ACETATE (ppm Mg)	0	0	150
OXYGEN ABSORBANCE (cc O <sub>2</sub> /g polymer) (10 days)	0.022	0.028	0.021
OXYGEN ABSORBANCE (cc O <sub>2</sub> /g polymer) (20 days)	0.024	0.024	0.023
OXYGEN ABSORBANCE (cc O <sub>2</sub> /g polymer) (31 days)	0.022	0.024	0.021
OXYGEN ABSORBANCE (cc O <sub>2</sub> /g polymer) (40 days)	0.025	0.027	0.024

6. Table 1 above sets forth the amount of oxygen scavenged over 10, 20, 31, and 40 days by samples of polyethylene terephthalate containing catalyst compounds according to the teachings of the '230 (Nichols) patent, compared to polyethylene terephthalate without the metal salt catalyst compounds. Sample 1 is polyethylene terephthalate polymerized by using sulfuric acid and no metal catalysts (control). Sample 2 is polyethylene terephthalate polymerized by using a catalyst system comprising antimony glycolate, cobalt acetate, and zinc acetate, such that the concentration of antimony in the polymer was 600 ppm, the concentration of cobalt in the polymer was 60 ppm, and the concentration of zinc in the polymer was 150 ppm. Sample 3 is polyethylene terephthalate polymerized by using a catalyst system comprising antimony glycolate, cobalt acetate, and magnesium acetate, such that the concentration of antimony in the polymer was 600 ppm, the concentration of cobalt in the polymer was 60 ppm, and the concentration of magnesium in the polymer was 150 ppm. These concentrations are at the high end of the ranges

taught by Nichols, (Col. 4, line 59 to col. 5, line 7) to most closely align with the present invention and to provide the samples with the best chance of scavenging oxygen.

7. As seen in Table 1, there is no oxygen scavenged by the catalyst compounds. The oxygen absorbance of the control sample (0.025 cc/gram polymer) is essentially identical to that of the test samples (0.027 and 0.024 cc/gram polymer, for samples 2 and 3, respectively) after 40 days, meaning the added metal salt catalysts had no effect in scavenging oxygen.

8. A gas chromatograph was used to measure the amount of oxygen scavenged by all of the examples given herein. More specifically, each vial was stored at room temperature for a measured amount of time. The contents of the vial were then analyzed by using a gas chromatograph that measures the ratio of nitrogen to oxygen in the vial vapor space as compared to a standard. The results reported in Table 1 were obtained by calculating the decrease in the oxygen in the vial vapor space as the oxygen was scavenged by the resin composition, and dividing by the amount of resin composition in the vial.

9. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6-11, 34, 36, 37, and 39-44 under 35 U.S.C. § 103(a) as obvious over Nichols, the '230 patent in view of Venkateshwaran et al., the '481 patent, or Blinka et al., U.S. Pat. No. 6,365,245 (the '245 patent). I respectfully disagree.

10. In my experience, I have observed that cobalt neodecanoate is not an oxygen scavenger. Cobalt neodecanoate is disclosed as a preferred catalyst in the '245 patent, and is a cobalt carboxylate, which is a type of compound preferred as a catalyst by the '230 patent. Table 2 below illustrates the lack of oxygen scavenging of polyester resin compositions comprising cobalt neodecanoate.

**TABLE 2**

Sample No.	Cobalt Neodecanoate (ppm)	Wet or Dry	Oxygen Scavenged After 21 Days (cc O <sub>2</sub> /g resin)
4	0	Dry	0.012
5	1150	Dry	0.009
4	0	Wet	0.012
5	1150	Wet	0.009

11. More specifically, Samples 4 and 5 were prepared as follows:

a. Dry samples

i. 3.3 grams of 12 % Cobalt TEN-CEM from OMG Americas Inc. (OMG CODE: 00084, Lot Number 59368), which contains 70% cobalt neodecanoate and 30% mineral spirits, was extrusion blended into 2000 grams of a 95/5 mole percent tere/napthalate PET copolymer of .84 Intrinsic Viscosity.

ii. The pellets were ground and placed into gas chromatograph vials.

b. Wet samples

i. Samples were prepared as above in step a.i.

ii. A small amount 0.001N Acetic Acid was added to wet the samples.

c. Oxygen analysis was done by gas chromatography, as described in paragraph 8 above.

12. Sample 4 contains 0 ppm of cobalt neodecanoate. After 21 days, only 0.012 cubic centimeters oxygen per grams resin (cc O<sub>2</sub>/g) was absorbed. This amount is well below any amount of oxygen that would minimally be absorbed by an oxygen scavenger, and is most likely attributed to variations in the volume of the gas chromatograph vials or the absorption of oxygen into the polyester. In any event, it

is not evidence of oxygen scavenging. Sample 5 contains 1150 parts per million cobalt neodecanoate. The amount of oxygen absorbed after 21 days is 0.007 (cc O<sub>2</sub>/g), no more than the comparative resin without cobalt neodecanoate.

13. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6, 11, 17, 34, 36, 37, 39, 43 and 44 under 35 U.S.C. § 102(b) as anticipated by, or in the alternative, under 35 U.S.C. § 103(a) as obvious over Konagaya et al., U.S. Pat. No. 5,434,000 (the '000 patent). I again respectfully disagree.

14. In my experience, I have observed that antimony trioxide and magnesium acetate are not oxygen scavengers. As shown in Table 1, Sample 3 contains substantially higher amounts of both antimony glycolate (antimony trioxide in ethylene glycol) and magnesium acetate than that taught by Konagaya, and yet exhibits no oxygen scavenging ability, as discussed above.

15. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6, 11, 30-34, 36, 37, 39, 43, and 44 under 35 U.S.C. § 102(b) as anticipated by, or in the alternative, under 35 U.S.C. § 103(a) as obvious over Kriesche et al., U.S. Pat. No. 5,565,545 (the '545 patent). I yet again respectfully disagree.

16. In my experience, I have observed that metal salts such as those disclosed as catalysts by Kriesche are not oxygen scavengers. As shown in Table 1, Samples 2 and 3 contain substantially higher amounts of antimony glycolate (antimony trioxide in ethylene glycol) than that taught by Kriesche, and yet exhibit no oxygen scavenging ability, as discussed above. Further, germanium dioxide is not capable of being an oxygen scavenger, because the germanium is already present in its highest oxidation state (+4).

17. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6-9, 11, 30-37, 39-41, 43, and 44 under 35 U.S.C. § 103(a) as obvious over Tindale, U.S.

Pat. No. 5,419,936 (the '936 patent) in view of the Aldrich Chemical Catalog, page 122 (1988). I respectfully disagree.

18. Under my direction, employees of the assignee of record who were not inventors of the subject application attempted to produce bottles according to the teaching of Tindale, *i.e.* by adding antimony oxide and reducing the antimony *in situ* with triphenyl phosphite. Following the patent exactly in terms of antimony and phosphorus concentrations, it was found that most of the antimony oxide was deactivated, either by reaction with the phosphoric acid to produce antimony phosphate compounds or by being reduced to the metal. There was insufficient catalytic antimony oxide left to adequately catalyze the melt polymerization and subsequent solid state polymerization. Nevertheless, Assignee's non-inventor research team was able to alter the method to polymerize the polymer and reduce the antimony as required in Tindale. More specifically, the phosphoric acid was omitted and the amount of antimony catalyst was increased such that, after 300 ppm antimony was reduced with triphenyl phosphite, there remained enough unreduced antimony to catalyze the polymerization. However, after 20 days, the oxygen absorbance of three samples prepared in this manner was measured to be 0.011, 0.005 and 0.003 cc O<sub>2</sub>/gram polymer. A control sample containing antimony only and no phosphorous acid provided an oxygen absorbance of 0.005 cc O<sub>2</sub>/ gram polymer after 20 days. These rates of absorbance of the oxygen are considered to be extremely low and do not show the presence of oxygen scavenging, particularly when compared to other oxygen scavenging particles which more typically have an oxygen absorbance of 0.5 cc O<sub>2</sub>/gram polymer or more after about 20 days. Indeed, these oxygen absorbances are lower than the Sample 1 from Table 1, which does not contain antimony.

19. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6, 11, 30-34, 36, 37, 39, 43, and 44 under 35 U.S.C. § 102(b) as anticipated by, or in the alternative, under 35 U.S.C. § 103(a) as obvious over Pengilly, U.S. Pat. No. 4,535,118 (the '118 patent). I respectfully disagree.

20. In my experience, I have observed that compounds of antimony and cobalt such as those disclosed by Pengilly are not oxygen scavengers because they are not capable of reacting with molecular oxygen. Again, reference is made to Samples 2 and 3 in Table 1, which contain antimony glycolate and cobalt acetate compounds in amounts greater than what is taught by Pengilly, and yet exhibit no oxygen scavenging ability, as discussed above.

21. I have also reviewed the Office Action dated February 24, 2003 and the pending claims, and understand that the Examiner has rejected claims 1-4, 6-9, 11, 30-34, 36, 37, 39-41, 43, and 44 under 35 U.S.C. § 103(a) as obvious over Pengilly, the '118 patent, in view of the Aldrich Chemical Catalog, page 122 (1988) and Venkateshwaran, the '481 patent, or Blinka, the '245 patent. I again respectfully disagree.

22. In my experience, I have observed that the compounds taught by Pengilly are not oxygen scavengers. Furthermore, Venkateshwaran '481 and Blinka '245 patents do not support the Examiner's position that metal salts and amines are inherent oxygen scavengers. The metal salts disclosed by the '481 patent do not support the Examiner's position, because they all contain metals in a less-than-fully-oxidized state, such as iron (II). These are *not* the type of metal salts disclosed by Pengilly as catalysts. The '481 patent does not disclose the compounds taught as suitable catalysts by Pengilly, nor does it suggest that a substitution of those compounds for the catalysts of Pengilly could be made.

23. Likewise, the '245 patent does not support the Examiner's position that the catalysts taught by Pengilly are inherent oxygen-scavenging particles. In Col. 4, lines 28-35, the '245 patent teaches two different oxygen scavengers. The first oxygen scavenger disclosed is ascorbate, which may be activated by a transition metal catalyst. In this system, the transition metal catalyst may be a metal, metal compound such as a salt, or a complex or chelate. Neither the '245 patent nor the incorporated references teach, disclose, or suggest that the metal compound such as a salt is or can be an oxygen scavenger, although they mention that complexes or chelates may be. In fact, the second oxygen scavenger disclosed is a transition metal



complex or chelate of a polycarboxylic or salicylic acid or polyamine. These scavenging compounds, however, are not similar in structure or function to the catalyst compounds disclosed by Pengilly.

24. In Col. 3, line 58 to Col. 4, line 7, the '245 patent discloses a catalyst in the form of a transition metal salt, that is used to catalyze the oxygen-scavenging ability of an ethylenically unsaturated hydrocarbon. There is no suggestion that the transition metal salt itself scavenges oxygen. Indeed, preferred catalysts are described as those that "readily interconvert between at least two oxidation states." Col. 3, lines 53-55. The catalysis of ethylenically unsaturated hydrocarbons is well known in the art and one of ordinary skill would readily recognize that the metal salt is the catalyst and not the compound reacting with the oxygen. Additionally, one of ordinary skill in the art would readily recognize that such catalyst compounds would not be effective oxygen scavengers because any reaction with oxygen that might take place would be reversible, *i.e.* the oxygen would be released as quickly as it may be reacted.

I declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the U.S. Code and that such willful false statements may jeopardize the validity of this application and any patent issuing thereon.

Respectfully submitted,

Edwin A. Sisson

Edwin A. Sisson

August 14, 2003